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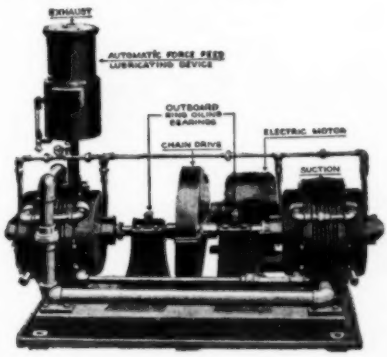

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OIL & FAT INDUSTRIES. Vol. V. No. 12. December, 1928. A monthly journal published by MacNair-Dorland Co. at 136 Liberty Street, New York, N. Y. Subscription, $3.00 the year, domestic; $4.00, foreign. Entered at the Post Office, New York, December 31, 1925, as second class matter under act of March 3, 1879.

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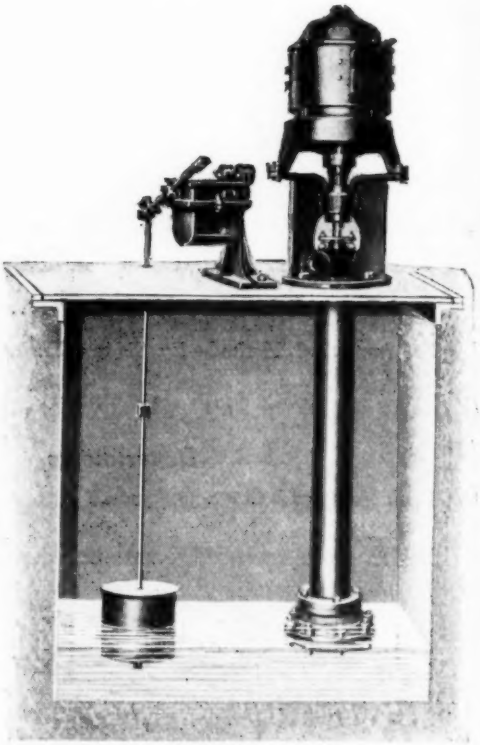

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OIL & FAT INDUSTRIES

VOLUME V DECEMBER, 1928 NUMBER 12

YOUR ADVICE - - - !

THIS issue of OIL & FAT INDUSTRIES, the last in its present shape and size, completes its fifth year of publication. Beginning next month, the magazine enters upon its sixth year in a larger size and in a broadened field of editorial activity. The commercial and business sides of the oil and fat industries, we feel, are in reality so closely related to the technical and scientific aspects of the industries, that a trade paper aiming genuinely to serve and represent such a group must of necessity give full consideration to all phases.

To the members of the American Oil Chemists Society, to other technical men, executives, purchasing agents, sales managers,—to the oil and fat industries as a whole, we want to say that the service which OIL & FAT INDUSTRIES can give, and the value of the publication to the various branches of the industries, will be directly proportional to the cooperation which we receive. The publishers want your advice, your suggestions, your criticisms. These are of inestimable value in the determination of editorial policies and in the general conduct of the publication. The more people who keep us informed of news, technical developments, and the like, and the more often they advise us, the better equipped we are to increase the value of the magazine to the industries.

Quite frankly, we want your advice. Write us if you have anything "on your chest." Drop us a line if you change your position, or put out a new product, or if you disagree with the stand taken in any of the scientific material published. In short, help us to give you and your industry a better trade paper.

—THE EDITOR.

Soaps from Organic Bases*

A Revolutionary Development in the Art of Organic Synthesis

BY R. B. TRUSLER†

THE combination, or chemical union, of a metal or basic group of any kind with a fatty acid produces a salt. If the fatty acid is one consisting of a relatively large number of carbon atoms, such a metallic salt is called a soap. The term "soap" in fact, is rarely applied to salts wherein the fatty acid contains less than nine carbon atoms, and in technology twelve carbon atoms are generally regarded as constituting the practical lower limit for soap-forming fatty acids. The most available soap-producing fatty acid is oleic acid. All grades of oleic acid on the market consist chiefly of true oleic acid together with isomers of apparently the same composition. The two solid fatty acids, stearic and palmitic acids, are next in abundance. Occasional special applications involve soaps made from linoleic acid and from lauric acid, and more rarely from numerous less available fatty acids that cannot be described here. In a general way, because of the indefinite gradation in the physical properties of the salts of the fatty acids, a soap-forming fatty acid may be defined as one whose sodium or potassium salt in water solution produces a foam when shaken. A more exact definition cannot easily be given, for it would involve consideration of the durability of the foam, the temperature, the concentration, and other conditions.

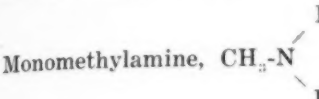
Salts produced by the combination of an organic base with a soap-forming fatty acid belong to the family of soaps just as correctly as do the salts obtained from any alkali or metal and the same fatty acid. An organic base may be described as a collection of non-metallic atoms arranged in a definite, invariable grouping, and which exhibit sufficient basicity to combine with an acid to produce a salt. The organic bases vary greatly in their basicity, i. e., in their combining power. For this reason some organic bases give stable compounds with only mineral acids and certain stronger (more highly ionized) organic acids, and do not combine appreciably with the soap-producing fatty acids. There are numerous organic bases, however, which combine with the heavy fatty acids to produce exceedingly interesting and important types of soaps.

Upon this occasion only the soaps made from organic bases containing one or more nitrogen atoms will be considered. These basic organic compounds are obtained mainly by synthetic chemical means through the substitution of some organic group in place of one or more of the hydrogen atoms normally attached to the nitrogen atom of the ammonia molecule. The replacement of one or more of the hydrogen atoms attached to the nitrogen nucleus makes possible the production of alkylamines and arylamines possessing marked basic properties. An alkylamine always is obtained when one or more of the hydrogen atoms attached to the nitrogen nucleus is replaced by

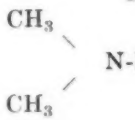
* Paper read at the meeting of the American Oil Chemists Society, October 25, 1928.

† *Industrial Fellow at the Mellon Institute.*

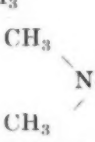
an alkyl, sometimes called aliphatic group. Hence the alkyl group methyl, CH_3-, occurs in



Monomethylamine, CH_3-NH$_2$,

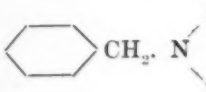


Dimethylamine, N-H, and

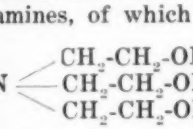


Trimethylamine, $N(CH_3)_3$

Similarly, the ethyl and propyl groups occur in the ethylamines and propylamines, etc. A single alkyl group may be shared between amine groups. A good example of this is ethylenediamine, NH_2. CH_2. CH_2-NH_2. Also, the alkyl group itself may have some grouping of atoms in place of one or more of its hydrogen atoms, as in the case of benzylamine,

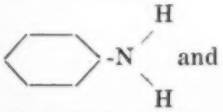


. Then there is the remarkable class of compounds known as the hydroxy-alkylamines, of which triethanolamine,

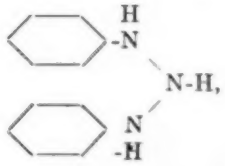


$N(CH_2$-CH_2-$OH)_3$, is an example.

The arylamines differ from the alkylamines in that an aryl, or aromatic, or a benzenoid group, replaces one or more of the hydrogen atoms. Aniline,



and diphenyl-guanidine,

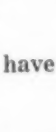


are representative compounds in this class of substituted amines.

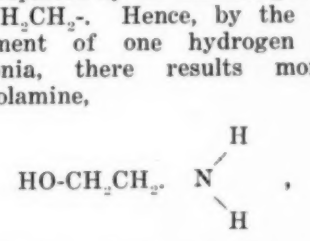
Ethanolamine Soaps
Theoretical Considerations

The soaps obtained from the ethanolamines promise to become outstanding among the organic base soaps, because of the peculiar properties of the ethanolamine fatty acid compound, as well as the striking and unusual character of these hydroxy-alkylamines themselves.

The ethanolamines, from which ethanolamine soaps are made, are three in number, viz., mono-, di-, and tri-ethanolamines. These compounds are synthesized from ammonia and fundamentally are substituted ammonia compounds, in which one or more of the hydrogen atoms of the compound



have been replaced by the ethanol group, HO-CH_2CH_2-. Hence, by the replacement of one hydrogen in ammonia, there results monoethanolamine,



which is chemically classified as a hydroxy-alkylamine. Since each of the hydrogen atoms can be replaced by an ethanol group, three ethanolamines are obtainable.

The chemical formulas and boiling points of, these ethanolamines are as follows:[1]

	Formula	Approximate Molecular Weight	Boiling Point
Mono-ethanolamine	$H-N(H)-CH_2CH_2OH$	61.07	171°C. at 757 mm.
Di-ethanolamine	$HN(CH_2CH_2OH)_2$	105.12	217°C. at 150 mm.
Tri-ethanolamine	$N(CH_2CH_2OH)_3$	149.16	277°C. at 150 mm.

The three ethanolamines differ slightly in their physical and chemical properties. They are miscible in all proportions with water, the ordinary alcohols, acetone and its homologues, glycerin, glycol, ethylene chlorhydrin and propylene chlorhydrin, and in general with many oxygenated organic compounds. Among the exceptions to this classification are ethyl ether and some of the aldehydes, in which the ethanolamines are scarcely soluble. Mono-ethanolamine is a colorless liquid, slightly viscous and possesses a faint but agreeable ammoniacal odor. It is one of the most hygroscopic substances known. Di-ethanolamine is also a colorless and odorless liquid. It is viscous like glycerin. Tri-ethanolamine, when pure, is a white, crystalline solid at room temperature. Other physical properties of the ethanolamines, including pH values, will be determined and reported in the future.

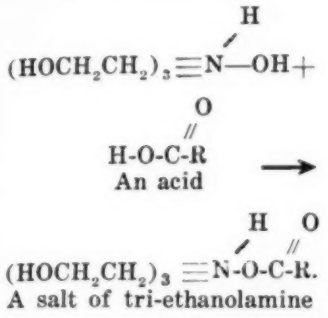
The ethanolamines are basic compounds, shown by their structural formulae. When dissolved in water, they are strongly alkaline to phenolphthalein. Titration with standard hydrochloric acid has shown that one molecular weight of any one of the ethanolamines is equivalent to one molecular weight of sodium hydroxide in combining with an acid. The tri-ethanolamine appears to be more basic than the di-ethanolamine, which, in turn, seems to be more basic than the mono-ethanolamine. The exact relationships will be revealed when the pH values are known.

The combination of any one of the ethanolamines with a fatty acid also results in a neutral compound. A study of the reaction has shown that, during neutralization of these hydroxy-amines, either in solution or in anhydrous conditions, the tertiary nitrogen becomes quaternary in the same fashion as the nitrogen atom in the ammonia molecule when neutralized under similar conditions. For example, in water solution it is evident that tri-enthanolamine is hydrated by adding one molecule of water, as

$$\left(\begin{matrix} HOCH_2CH_2 \\ HOCH_2CH_2 \\ HOCH_2CH_2 \end{matrix} \!\!\rangle\!\!-\!\!\rangle N\text{-}H \right) OH$$

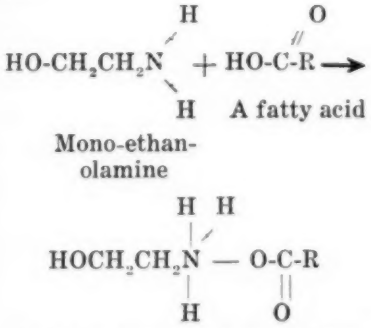
[1] These boiling points were determined by the Carbide and Carbon Chemicals Corporation's Multiple Industrial Fellowship at Mellon Institute.

This combines with an acid in the same manner as ammonium hydroxide, as follows:



A salt of tri-ethanolamine

The reaction between anhydrous ethanolamine and an anhydrous acid yields the same result, since the nitrogen atom again changes valence. It has been found that a molecular equivalent of any one of the ethanolamines combines with exactly one molecular equivalent of an organic acid; and if the organic acid is one of the soap-producing fatty acids, the resulting product is a soap. The reaction may be illustrated by the following equation:



A soap of mono-ethanolamine

The compounds made in this manner were found to be anhydrous. The absence of any water of reaction showed that there was no formation of esters by the acid condensing with the hydroxyl group.

Preparation of Ethanolamine Soaps

In the course of his researches Koganei[2] found that mono-ethanolamine (or beta-amino-ethanol) was sufficiently basic to react with stearic and oleic acids, to produce compounds having decided soap-like properties. This work was done when mono-ethanolamine was prepared by tedious academic means. A few years later, when the ethanolamines were approaching commercial availability through a new chemical synthesis, the preparation of all types of their soaps on a large scale became possible, thus permitting a study of them and their technical applications[3].

There are three possible homologous types of ethanolamine soaps, depending upon which one of the three ethanolamines is used. However, for technical purposes, a mixture of the three can be used advantageously, because they are quite similar in their properties. This mixture, which consists of approximately 75 to 80 per cent tri-ethanolamine, 20 to 25 per cent di-ethanolamine, and 0 to 5 per cent of mono-ethanolamine, has a price advantage over any one of the constituents in that there is no additional cost for fractionation, which, of course, would be necessary if the individual components were required separately. The mixture of these homologues is therefore offered to the trade under the name of "Ethanolamine." While we are interested in the soaps from the individual ethanolamines themselves, we are most interested in the soaps involving the technical ethanolamine, which, incidentally, is obtainable in a high state of

[2] Koganei, Biochem. J., 3, 19 (1923).

[3] The writer was preceded in this study by Dr. O. F. Hedenburg, another Industrial Fellow of Mellon Institute.

purity. Any one of the three ethanolamines as well as technical "Ethanolamine" combines readily with fatty acids to produce soaps. In order to prepare any one of the ethanolamine soaps, it is best to determine the basicity of the ethanolamine by titration in water against a standard acid. This procedure will allow the calculation of the concentration of the particular hydroxy-alkylamine and also takes into account the presence of moisture. The acidity, or combining value, of the fatty acid should also be determined according to a standard method for titrating a fatty acid in alcohol. After these two values have been found, weighed quantities of both ingredients may be combined. It would be convenient, indeed, if approximately correct quantities of both ethanolamine and the fatty acid could be run into the reaction kettle, and then the final necessary amount of either ingredient could be added to bring about a neutral product, as shown by testing portions of it by an indicator. But this method so far has proved to be unreliable, because even with methyl red in alcohol the color change from red to yellow occurs so slowly that uncertain results are had.

The liquid fatty acids, such as oleic acid, permit the easy preparation of ethanolamine soaps. For example, let us consider the preparation of ethanolamine oleate from the technical oleic acid, which may be the "saponified" or the "distilled" variety. Analyses of the oleic acid to be used have shown it to have an acidity of 97.5 per cent in terms of oleic acid, and the ethanolamine has been found to have such alkalinity that 100 grams will combine with 215 grams of pure oleic acid[4]. Then, for every 100 Kilos of ethanolamine there must be added $\frac{215}{97.5}$ or about 220.511 Kilos of oleic acid. The oleic acid is run into the container, which should be provided with a strong and slowly revolving stirrer. The entire amount of ethanolamine can be run in before stirring is begun. When stirring is finally started and the two ingredients are brought into intimate contact, reaction occurs with sufficient heat to keep the contents mobile during stirring. External heat does not promote the combination. Inasmuch as neither of the components appreciably vaporizes, there is no loss during the reaction. Water increases the viscosity of these soaps, and hence the ethanolamine should not contain over 10 per cent moisture, and preferably should be nearly anhydrous, otherwise stirring the reaction mixture will become more difficult. The writer wishes to mention again that the two ingredients unite exothermically, showing a decided chemical reaction.

Combining solid fatty acids, such as stearic acid, with the ethanolamines requires slightly different treatment. In order to mix intimately a solid fatty acid with an ethanolamine to bring about complete reaction, two different procedures are available, namely: (1) The solid fatty acid may be melted and heated to about 60°, and into it is stirred the correct quantity of ethanolamine, which has also been warmed to about 60°. This preheating plus the heat of reaction

[4] While the molecular weight of oleic acid is about 282.27, and the molecular weight of triethanolamine is about 149.16, it has been mentioned that technical ethanolamine contains some of the lighter molecular weight ethanolamines, which increase the ratio of oleic acid to ethanolamine above the molecular proportions of 282.27 to 149.16.

will keep the soap sufficiently soft to allow thorough stirring, otherwise the viscous product will solidify. (2) the stearic acid, or other fatty acid, may be dissolved in a solvent, such as denatured alcohol, to which the ethanolamine is then added with stirring. The soap may be recovered by evaporating the solvent.

Physical Properties of Ethanolamine Soaps

The consistency of any one of the ethanolamine soaps when anhydrous or nearly anhydrous, depends more upon the nature of the fatty acid employed than upon the particular ethanolamine. The oleate of mono-ethanolamine has the consistency of petroleum jelly, while the oleates of di- and tri-ethanolamine are only slightly more viscid. Compared to these, mono-ethanolamine stearate is a hard, wax-like solid.

The soaps prepared from ethanolamines are colored pale yellow to reddish brown, depending upon the purity and color of the fatty acids employed. Generally speaking, all these compounds have an agreeable, soapy odor.

The soaps made from ethanolamines and the higher fatty acids, such as oleic and stearic acids, are soluble in a great variety of organic solvents, which is one of their unusual and outstanding properties. They are dissolved readily by benzene, toluene and similar compounds. Turpentine, alcohols, glycol, glycerine, ketones and many aldehydes are excellent solvents for these soaps. It is surprising to note that even heavy petroleum products, such as lubricating oils and petrolatum, will dissolve considerable amounts of these soaps. In most cases, excepting the heavy oils, the ethanolamine soaps are soluble in the solvents in all proportions, yielding transparent solutions.

Dilute water solutions of the ethanolamine soaps are opalescent to milky, resembling somewhat sodium soaps. The oleate in water froths readily and maintains its soapy character upon standing, but the stearate and, to a less extent, the palmitate slowly revert to the original components, with subsequent partial separation of the fatty acid.

Ethanolamine oleate is capable of taking up a considerable amount of water and remaining jelly-like. The addition of water first causes an increase in the consistency of the soap, this effect continuing until the weight of the water absorbed is about equal to the weight of the soap. Further addition of water renders the soap more plastic and somewhat creamy.

Ethanolamine stearate behaves differently than the oleate upon hydration. Upon the absorption of water, the hard wax-like soap softens, becomes opalescent and reminds one of an agar-agar gel in appearance and consistency.

Applications of Ethanolamine Soaps

The study of the applications of the ethanolamine soaps has chiefly concerned the oleates because of their ease of handling. These oleates are nearly liquid soaps and have comparatively high stability in the presence of water. The two classes of soaps, those from liquid fatty acids and those from solid fatty acids, are not interchangeable in their applications, each one having rather well defined adaptations.

Ethanolamine Oleate as an Emulsifier.—Ethanolamine oleate possesses striking emulsifying ability

in promoting oil-in-water emulsions. These emulsions can be made in any one of the known technical ways, but a very simple and efficient method may be employed. The amount of ethanolamine oleate required for the emulsification of an oil depends largely upon the nature of the oil involved. Some oils emulsify with ease as compared to others; for example, linseed oil is much more readily emulsified than olive oil, and therefore requires less soap. This same difference has been observed in the case of mineral oils. In a general way, however, it may be said that from 6 to 8 per cent of soap on the basis of the oil used is sufficient for making a good emulsion. The oil to be emulsified is weighed out in a suitable container. About six to eight parts of ethanolamine oleate per hundred parts of oil are dissolved in the latter during warming and stirring. A transparent or nearly transparent solution of soap in oil results, which may be stored for a stock from which emulsions may be prepared as needed. In order to prepare the emulsion, it is only necessary to add the desired amount of water in the following manner. Supposing an emulsion of 80 per cent of olive oil in water is desired. Water is slowly stirred into the oil containing the dissolved soap. At first the liquid becomes cloudy and somewhat thicker. Further addition of water with stirring will bring about a creamy paste, and when about ten to fifteen parts of water have been added a creamy emulsion results. The balance of the water may be quickly put in. This will produce a heavy, quite permanent emulsion that can be diluted to any desired concentration. In this way a concentrated stock emulsion may be kept on hand from which thinner emulsions can be made when required.

Vegetable oil emulsions of this nature appear to have many applications for non-edible purposes. Excellent emulsions for textile oiling and fiber lubrication have been prepared from olive and palm oils.

Mineral oils may be similarly emulsified. Usually a little more soap is required than for vegetable oils, but the necessary amount can readily be found by experimentation. Mineral oil emulsions of this nature have shown some desirable features for cutting and grinding purposes.

Owing to the solubility of the ethanolamine soaps in petroleum products, some very interesting special applications have been tried on an experimental scale. It has been found that small amounts, in the proximity of 0.3 to 1 per cent of these soaps, dissolved in lubricating oils, increase their viscosity to a great extent. Oils are thus rendered non-running and become suitable for pressure oiling systems (not for internal-combustion engines) and as cup and gear greases.

A convenient and efficient means for removing oil and grease from articles and clothing can be had by dissolving about 10 to 20 per cent of the oleate in a convenient solvent, such as naphtha, carbon tetrachloride, and the like. A solution such as this has been successfully used in cleaning greasy walls, woodwork mechanisms, automobile bodies, and parts covered with grease and oil by brushing or rubbing it over the surfaces to be cleaned. After the solvent has evaporated, the grease and oil can be often completely removed by washing with water, because the grime is removed as an emulsion as the soap is taken up by the water. Ethanolamine oleate in

carbon tetrachloride (or ethylene dichloride) will be found useful in removing grease spots from garments. Before the fabric is wetted, the grease spot is moistened with the ethanolamine soap — carbon tetrachloride solution. This will penetrate grease, fresh paint and the like; and after the solvent has evaporated, the application of water with a little rubbing will emulsify the oil content of the stain so that it can be removed.

By far the most important application of ethanolamine oleate seems to be its adaptability for dry-cleaning fabrics and wearing apparel. While this study is still in the experimental stage, sufficient satisfactory observations have been made over a period of several months to warrant the prediction that the soap will be technically valuable. The fact that ethanolamine oleate is readily soluble in naptha over a wide range of concentrations is a feature that makes it especially important, since no special formulas are required in preparing a dry-cleaning soap and it is not necessary to employ an excess of the fatty acid to keep the soap in solution as is the practise for sodium, potassium, and ammonium soap compositions generally used for dry-cleaning purposes. This implies a corresponding decrease in the development of rancid odors in garments after cleaning, since there is no excess fatty acid to be rinsed out. The soap remains in solution in the solvent in the presence of small amounts of moisture which is a desirable feature for drycleaning soaps[5]. For cleaning felt and wool hats where the soap in naphtha is applied particularly good results have been obtained. The naphtha solution of ethanolamine oleate exhibits the striking characteristic of foaming or frothing much like water solutions of sodium soaps, though, of course, to a less extent.

Other Suggested Uses for Ethanolamine Soaps.—Inasmuch as ethanolamine is a surface tension depressant of real merit, it is possible that ethanolamine oleate can be employed as a wetting out agent, or even as a surface tension depressant for special needs.

Experience has shown that ethanolamine has unusual fiber penetrating power, especially in water solution. In order to render this penetrating action available, so that the compound may be used as an introfier, or carrier, in nonhydrous, oily systems, it is suggested that the oil-soluble ethanolamine oleate be used for this purpose.

The excellent emulsifying ability of ethanolamine oleate has been described. It is suggested that this feature be applied for the preparation of concentrated emulsions for veterinary purposes, such as cattle sprays, and poultry, house disinfectants and insecticides. Experience has thus far shown ethanolamine soap to be non-toxic and non-irritating for external use.

Water suspensions of clays and of other finely divided minerals have been found to be stabilized by the addition of small amounts of ethanolamine oleate. Ceramic uses are therefore suggested. A similar application might be made in the case of oil paints. It may be found that a small amount of the anhydrous soap dissolved in the oil would render a more permanent suspension of the pigment, or perhaps aid in preventing the pigments from forming a dense layer in the bottom of the container as they settle out.

The observation of the penetrating and softening action of ethano-

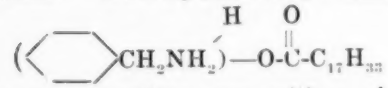
[5] Hatfield & Alliott. J. Soc. Dyers Colorists, 44, 170 (1928).

lamine upon hair led to the preparation of shaving creams with this soap replacing part of the sodium and potassium soaps. The technology of shaving creams involving ethanolamine soap has not been worked out, but the experience to date suggests the use of ethanolamine oleate in creams that are not excessively superfatted.

Other Alkylamine Soaps

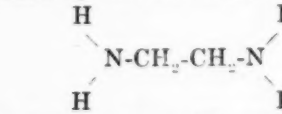
Simple alkylamines, such as the methyl and ethylamines and benzylamine, combined with fatty acids to form soaps[6]. Ethyl and methylamine soaps have properties nearly identical with the corresponding ammonium soaps.

Benzylamine readily forms liquid to viscous soaps with the fatty acids. Benzylamine oleate

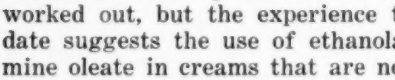


is a pale yellow soap with a viscosity like glycerin. It has a peculiar but agreeable odor, somewhat resembling nasturtium. This soap is peculiar in that it appears to be as readily soluble in oils and organic solvents as it is in water. It foams in water and allows the preparation of good emulsions. At present the cost of benzyl-, ethyl-, and methyl-amines is far too great to permit their use in the manufacture of commercial soaps.

Ethylene diamine, a double amine whose composition is represented by the formula,



has both commercial and technical possibilities. The synthesis of the compound has been accomplished by an improved process which will permit commercialization when uses are found for it. As the formula shows, it can combine with one or two fatty acid molecules. One amino group is decidedly basic; but after combining with a fatty acid to give a mono-basic soap of



the second amino group forms stable compounds only with the stronger fatty acids.

The mono-oleate and mono-stearate of ethylene-diamine are strongly basic in water solution and froth readily. They are soluble in alcohol, but poorly soluble in petroleum products and in benzene and its homologues. A highly basic, non-metallic soap, such as either the oleate or stearate of ethylene-diamine, is suggestive of desirable technical applications. It should be mentioned that the mono-stearate of ethylene-diamine is crystalline and flakey, while the two oleates are viscous liquids.

Soaps from Arylamines

Arylamines in general combine with fatty acids to form soaps. This comparatively new field is yielding products of unusual interest and importance. Attention should be called to the soaps of the cinchona alkaloids[7], which are commercially known as "Konate." These cinchona soaps, which are soluble in petroleum distillates and in various other organic solvents, have been found to be most efficacious agents for mothproofing fabrics and furs. The study of many arylamine soaps, their nature and application, is now being undertaken by the writer and the results will be reported later on.

Resumé

Soaps have been made from fatty

[6] Gibbs, J. A. C. S. 28, 1410 (1906).

[7] Jackson and Wassell, Ind. Eng. Chem., 19, 1175 (1927).

acids and organic bases containing nitrogen. The soap-forming bases are alkyl substituted amines and aryl substituted amines. In this paper alkylamines have been especially considered. Among them the hydroxy-alkylamines known as the ethanolamines have outstanding importance.

Ethanolamine soaps have diversified properties which may later bring them into technical importance. Among the prospective uses are the following:

(1) They are excellent emulsifiers for vegetable, animal and mineral oils.

(2) They are thickeners for lubricating oils.

(3) Incorporated in special compositions, they have detergent and cleaning utility.

(4) The oleates are efficient dry-cleaning soaps, due to their solubility in organic solvents and to their specific detergent action.

(5) Their great solubility in almost all liquids will undoubtedly eventuate in other industrial applications.

New Oilseed Crushing Mill in Norway

According to the Norwegian Trade Journal, the Nordiske Fabriker, De-No-Fa A/S have decided to erect an oilseed crushing mill in connection with their works at Fredriksstad, Norway.

This firm is the principal operator in the international whale oil pool. It is considered that, in conjunction with their existing mills at Oslo and Stavanger, the new mill will be able, not only to satisfy the requirements of the Norwegian market in regard to vegetable oil, but will also be able to export such oils. The share capital of the De-No-Fa will be enlarged by the issue of three million kroner. It is stated that this amount has already been guaranteed by the principal shareholders.

Olive Oil Plant Enlarged

Work recently began on a forty by forty-five foot, two story tile storage building to be added to the Rossville (Cal.) olive oil factory in order to care for the 1928 crop. Three years ago when Charles Livoti started the production of olive oil the present factory produced 4,000 gallons of oil. Two years ago the output was doubled, and last year it amounted to 35,000 gallons. With 1928 crop indications excellent the Rossville factory expects to utilize all the additional space being provided.

New Process for Sulphonating Oils, England

A British patent has just been issued to E. C. R. Marks of London on behalf of the Chemische Fabrik Stockhausen et Cie., Grefeld, Germany, wherein oils, fats or fatty acids or mixtures thereof are sulphonated in the known manner and after removal of the dilute acid, the resulting product is mixed with water or dilute acids and allowed to stand until no further separation takes place. A neutral or indifferent solvent such as trichlorethylene may be added before, during, or after the sulphonation, whereby two layers are obtained, one containing the unattacked or slightly attacked component and the other, sulphuric acid esters. The reaction is preferably carried out with increased quantities of concentrated sulphuric acid at a temperature below 150° C. Examples of the process are given in which castor oil is sulphonated.

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The Hydrogenation of Soybean Oil

Experimental Investigation of Its Application to Lard-Substitute Production

BY AUGUSTUS H. GILL AND YU M. MA.

ALTHOUGH there are four varieties of soybean, only one, the yellow, or soybean as it is universally called, is used in the soybean mills. The mills, or soybean oil-pressing plants, rank foremost in the manufacturing industry in Manchuria which comprises the three Provinces: Fengtien, Kirin and Heilungkiang, having 20 million inhabitants and an area of 365,000 square miles. The output of the oil in 1923 was about 500,000 tons. In Dairen, the center of soybean mills, 390 tons of oil are produced daily.

Since soybean oil is between peanut and cottonseed oils in the uses and the qualities, and no hydrogenation plant is established in China, the commercial hydrogenation of this oil, especially for edible purposes, should be developed, to better feed the 400 million Chinese, as well as their neighbors of non-farming nations.

The oil used had the following characteristics:

Specific gravity at 15.5°	0.925
Free fatty acid calculated as oleic acid	0.57%
Iodine number	140.0

The catalyst is 'poisoned' by free fatty acids and moisture in oil. The latter is removed by drying and the former with caustic soda solution.

The oil was agitated with an electrical stirrer and 80 percent of the theoretical amount of caustic soda solution (16° Be.) were added. After 30 minutes the oil was heated in a water bath to 50° until the emulsion was "broken." It was then allowed to settle and was filtered through filter paper. The oil thus obtained was dried in the same cup for ten minutes at 120°, being agitated during the operation and then being filtered again. It was stored for hydrogenation.

The hydrogenation took place in a bronze autoclave holding about 600 cc., provided with a stirrer and oil bath for heating.

Preparation of the Catalyst

There are two distinct methods of preparing the catalyst, one being known as the dry process and involving nickel salts in the dry state, and the other being known as the wet process, according to which the catalyst is prepared by reduction in oil. The latter process has been proved simpler and more reliable.

The catalyst was prepared by grinding nickel formate, the most commonly used material in the wet process, in the oil, and heating the oil emulsion thus obtained in the autoclave, hydrogen being passed in to displace the air in the autoclave until the reduction was complete. After the air had been completely displaced, (usually in five minutes), the burners were lighted and so regulated that the temperature was raised gradually and kept at 240°. When the bubbles of hydrogen became even and regular, usually after three to four hours, the nickel formate was considered to be completely reduced. The catalyst was now ready for use. It was newly prepared for every hydrogenation.

Hydrogenation

The oil to be hydrogenated was poured into the autoclave, the air displaced as before and the cap bolted on. The burners were lighted and regulated so as to get a definite temperature, and the valve admitting hydrogen was regulated so as to get a definite pressure indication on the gauge. Stirring was begun and after a definite time a sample of 20 to 60 cubic centimeters was withdrawn with a pipette, and filtered through a hot water funnel. The process from air-displacing to sample-filtering was repeated until several samples had been taken.

The conditions under which the oil was hydrogenated and the iodine numbers of the samples were as follows:

Run 1. Temperature 170°C., pressure 40 pounds, 100 revolutions per minute, 0.6% catalyst.

Sample	Elapsed Time Minutes	Iodine No.
1	60	106.0
2	120	95.7
3	180	87.3
4	240	79.0
5	300	75.8
6	340	72.0
7	370	66.8

Run 2. Temperature 180°C., pressure 40 pounds, 100 r.p.m., 1.00% catalyst.

Sample	Elapsed Time Minutes	Iodine No.
1	60	83.9
2	120	72.7
3	180	66.4
4	240	62.9
5	300	56.8

Run 3. Temperature 170°C., pressure 2 pounds, 406 r.p.m., 1.00% catalyst.

Sample	Elapsed Time Minutes	Iodine No.
1	60	2.78
2	70	2.54

Run 4. Temperature 170°C., pressure 20 pounds, 406 r.p.m., 1.00% catalyst.

Sample	Elapsed Time Minutes	Iodine No.
1	15	18.3
2	20	18.0
3	25	17.3

Run 5. Temperature 160°C., pressure 10 pounds, 400 r.p.m., 0.40% catalyst.

Sample	Elapsed Time Minutes	Iodine No.
1	15	45.0
2	30	42.2
3	45	37.9

Run 6. Temperature 150°C., pressure 10 pounds, 400 r.p.m., 0.40% catalyst.

Sample	Elapsed Time Minutes	Iodine No.
1	10	78.2
2	20	66.6
3	30	50.2

Run 7. Temperature 150°C., pressure 10 pounds, 320 r.p.m., 0.40% catalyst.

Sample	Elapsed Time Minutes	Iodine No.
1	30	82.1
2	60	56.6
3	90	43.9
4	120	40.5

The Effect of Pressure

The working pressure[1] of hydrogenation of cottonseed oil is below 40 pounds. No higher pressure seems to be necessary for hydrogenation of soybean oil. Moore[2] has pointed out that the hydrogenation rate of cottonseed oil is approximately proportional to the pressure. This fact will hold true for soybean oil.

[1] Rogers "Industrial Chemistry" (1926) 764.
[2] Jour. I. and E. Chem. 9, 459 (1917).

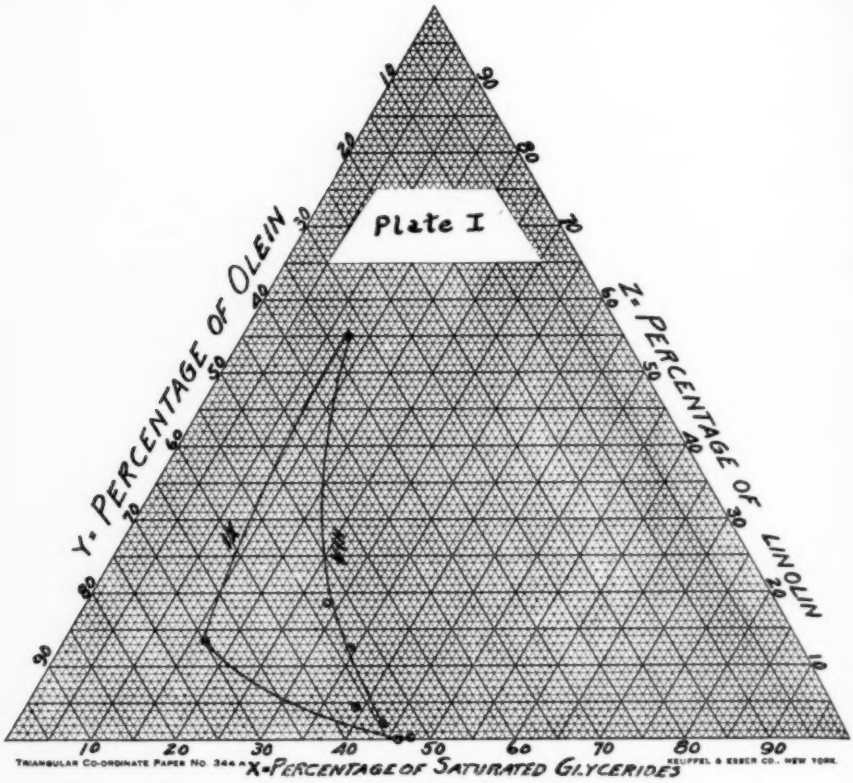
Plate I
Y- PERCENTAGE OF OLEIN
Z- PERCENTAGE OF LINOLIN
X-PERCENTAGE OF SATURATED GLYCERIDES
10 20 30 40 50 60 70 80 90
TRIANGULAR CO-ORDINATE PAPER NO. 344-A
KEUFFEL & ESSER CO., NEW YORK.

Graphs showing progress of reaction in hydrogenation of soybean oil

The Effect of the Amount of Catalyst

As in practically all catalytic reactions, the hydrogenation rate[3] is increased as the percentage catalyst is raised. The fact[4] that no more than one percent of catalyst is necessary, is applicable to the hydrogenation of soybean oil, for no more than one percent of catalyst was used in the experiments and the results were satisfactory.

The Composition of Fats from Runs 6 and 7

The fats were analyzed in order to know how the reactions progressed in the course of the hydrogenation process. The Gusserow-Varrentrapp lead-salt-ether method was used here. It is in principle a separation of two groups of lead salts of fatty acids based on the difference in solubility of the two groups in ethyl ether. The detailed procedure of analysis has been fairly well mapped out by Tortelli and Ruggieri[5] and others.

The composition of the fats, together with the iodine numbers of the fats and the liquid fatty acids, were as follows:

[3] Rogers, ibid.
[4] Moore, ibid.

[5] Lewkowitsch Analysis Oils, Fats and Waxes, I 560 (1921).

		Saturated Glycerides x	Per Cent Olein y	Linolin z	Fat A	Iodine Number Liquid Acids B
Run 6	(1)	28.5	53.5	18.0	78.2	114.1
	(2)	34.2	53.7	12.1	66.6	100.0
	(3)	43.4	54.5	2.1	50.2	92.7
Run 7	(1)	16.5	70.0	13.5	82.1	103.5
	(2)	39.0	56.2	4.8	56.6	97.3
	(3)	46.3	53.7	0.0	43.9	86.2
	(4)	47.5	52.5	0.0	40.5	80.3

Graphs I and II, plate 1, show the composition of the fats during hydrogenation; taking the composition of the oil as x, (saturated glycerides) 12.5% y, (olein) 33.4% z, (linolin) 54.1%.

Both the figures show that linolin changed into saturated glycerides and, more quickly, into olein. The linolin disappeared when the saturated glycerides reached 45.6 percent, then olein changed into saturated glycerides very slowly. The agitation was higher in run six than in run seven, so the reaction was quicker in the former than in the latter.

The Melting Points of Fats from Run 7

As the hydrogenated fat is to be used for edible purposes, its melting point should approximate that of lard. It was determined with the Mulliken capillary tube apparatus[6]. The melting points of the fats, together with the corresponding iodine numbers were as follows:

Melting points °C	18.5	35.0	39.1	46.9
Iodine numbers..	82.1	56.6	43.9	40.5

The melting points of lard, "Crisco," and "Snowdrift" were determined with the same apparatus. They are 40.4°, 40.7°, and 42.3° respectively; 40° is taken as the melting point of the commercial hydrogenated soybean fat.

Conclusions

(1) An increase of temperature, pressure, agitation or amount of catalyst will increase the hydrogenation rate.

(2) In high agitation, at 320 r.p.m. or above, the change of 10° in temperature between 150° to 180° produces little or no effect.

(3) The hydrogenation rate is approximately proportional to the pressure, the amount of catalyst, and one half of the speed of agitation.

(4) No more than one per cent of catalyst is necessary.

(5) The course of the reactions of hydrogenation of soybean oil is the same as with cottonseed oil.

(6) The commercial hydrogenation of soybean oil can be a reality, for it is easily hydrogenated. As in run seven the time for obtaining the fat of 40° melting point was only one and one-half hours, although the time may be different on the commercial scale.

[6] Woodman "Food Analysis" (1915) p. 154.

United States Bureau of Mines, Reno, Nev., has recently finished a study of native clays to be used as substitutes for fullers' earth in bleaching oils. The raw clays studied resemble sepiolite, and the clays requiring acid treatment resemble bentonite.

The Use of Fats in Baking*

Various Types of Baked Goods Present Different Shortening Problems

BY M. B. GRAFF†

THE chemistry of the fats and oils is an old subject to us. The methods of manufacture and refining, of packaging and distribution, are familiar to all of us. Some of us possibly have an idea that we know something of the use of fats in soap making and are well versed in the values and properties of the fats for this purpose.

The soap making industry is an old one, but so is the baking industry. Soap making apparently passed from the position of an art to that of an organized industry, and then to a science, long before baking began to take the same trend. Recent years have seen baking change from an art to a tremendously organized business. We watch its present struggle to get organized on a scientific basis. In the baking industry today the terms "scientific, technical, research and investigation" are flung about with the utmost abandon.

Flour Most Important

In baking, wheat flour is unquestionably the basic ingredient and one of first importance. Hence, the very important and advantageous position of the cereal chemist in the industry. At the same time, the fats play an important part in most baked goods—to say nothing of sugar, milk, eggs, yeast, baking powder or other chemical leaveners, etc. In these days of specialization, a cereal chemist is not supposed to know much about fats. As a result, the interest of the fat and oil chemists has been developing in this interesting field.

It is our purpose to touch upon some of the problems presented by the use of fats in some varieties of baked goods—and there is a large field—rather than to undertake to present the solution of any of the problems. As this is the first paper of it kind presented to this society, it is more important to get a view of the field.

It is possible to present a fairly clear classification of bakery products, but we prefer to take them up in a fairly logical order and discuss each one as we go along.

Shortening in Bread

Take the production or manufacture of bread. This is the branch which has become the most highly organized, the most highly mechanical of any bakery line. The raw ingredients are introduced at one end and the wrapped loaf delivered at your home with scarcely a touch of human hand, and under remarkably well controlled conditions. The quantity production is almost appalling.

Fat in bread, however, is a very minor ingredient. It is used in the proportion of only a few per cent of the flour. Two and one-half or three pounds to 100 pounds of flour, with a few pounds of milk, malt, salt and yeast and some 60 pounds of water will make up a bread dough.

Bread is judged or scored organoleptically. Reduce the shortening below 2½ percent or raise it over 3 percent and an expert scorer, on a number of loaves of each,

*Presented at Second Fall Meeting, A.O.C.S., New York, Oct. 26.

†*The Procter & Gamble Laboratories.*

will give an average lower score on either side of those limits, other conditions remaining the same.

You will say this should be easily solved. So it should, but with varying shop conditions, varying temperature, humidity and time of fermentation, varying flours and formulæ, and varying tastes and judgment, all of it has not yet been completed.

Pie Baking Difficulties

Take another class of baked goods, the great American pie. Although 80 percent of the bread baked in our country comes from the bakery, a much smaller percent of pies come from the bakery. There are three reasons. Some bakers cannot bake a good pie, some bakers will not bake a good pie, and those bakers who do make good pies have difficulty in overcoming the effect of the other two classes and the difficulties of distribution. Nevertheless, pie baking is going on in some very large units.

What are some of the problems connected with making a good pie crust? Of course, here again comes in the question of what is a good pie crust. But putting that aside, we have a problem.

If we mix oil with flour in ordinary pie crust proportions, say one of fat to two of flour, we get a soggy mass which will hardly take up enough water to make a dough or a good crust.

If we take the plastic fats, we may by proper manipulation get several varieties of tender pie crust. There is nothing very complicated about it, and a little care in handling, and sometimes of temperature control, will always give a tender edible crust.

A characteristic of flour, however, is very evident in this case. The tendency for the gluten to develop when wet with water and handled, and in this way produce a tough crust, is a serious problem. Balancing the character of flour, the character and quantity of shortening, the quantity of water and time and temperature make an interesting study.

Pie crust, with its simple composition and its simple manipulation, should be a good place to study shortening values. Its susceptibilities to slight variations in handling and baking, and the difficulty of measuring the effects after baking greatly reduce its possibilities.

Similar to pies, but influenced still more by the physical manipulation, are the pastries. These products indicate to us that the effect of shortening is the separation of the strands or layers of gluten and other body forming materials to keep them from matting together into a solid mass. The patty shells and puff pastry, and the Danish pastry rolls and material of that kind are the result of developing a gluten mass and then rolling it out in thin sheets alternating with layers of fat.

The physical properties of the fats are of great importance in producing the right kind of pastry and pie shells. The melting point has much to do with the edibility of the pastry. The chemical composition and the care used in preparation have much to do with its flavor. An off flavored fat or one which readily oxidizes in handling or baking will have a very bad effect on the finished product. We have discussed only the crusts and not the fillings of pies and pastries.

Cakes and Cookies

Turn to another line of bakery products known as sweet goods. This includes cakes and cookies and other products containing a noticeable quantity of sugar.

In these doughs we have much more complicated formulæ or systems. Besides the flour, the sugar, eggs and milk play an important part rather than being just minor ingredients. The sugar is frequently equal to the flour, and the eggs and milk at least half as much. With other material, such as molasses, glucose, invert sugar, cocoa or chocolate, and occasionally glycerin, there are many factors to evaluate.

The use and function of the shortening in most of these is of very great importance. Some of them require more than others.

We have the rich heavier cakes of the pound cake type in which originally the fat or butter was about one-fourth of the dough. This is generally cut now so that the shortening is only about half as much.

Strange as it may seem, in this type of cake the shortening is also used to produce a leavening effect. If the shortening has the proper consistency and is properly used, it is possible to incorporate into the shortening-sugar mass of the mixture sufficient air finely distributed throughout the mass to give the rise, expansion or volume required. This means the lightness of the cake. The beating of the eggs carries in some air, but most of it is incorporated with the fat. It seems probable that the production of a light aerated mass of sugar and shortening produces a very fine cellular structure of all the ingredients in the cake dough. When subjected to the heat of the oven all of the ingredients expand, but chiefly the moisture and air in the fine cells cause the dough to swell. If the formula is properly balanced and the temperature is properly regulated, the eggs and flour will coagulate when the maximum expansion has taken place and the cake retains its size, shape and internal structure. There are several of these qualities by which the cake is judged. After its external appearance is the smoothness and evenness of its grain and texture when cut. Upon these depend its tenderness and edibility.

A very simple and yet edible cake can be made by creaming two parts of sugar with one of shortening, adding one part of whole eggs to this mass, preferably before the creaming is finished, then stirring in one part of skim milk and two parts of flour. A little salt and flavoring completes the dough which bakes to a very nice, light, tender cake.

From this kind of a cake the formulæ grade down with less and less fat into the class of sponge cake with very little or no fat at all. The importance of the fat as a carrier of air becomes less and its function is more simply that of a shortener or to produce tenderness. The baker takes advantage as far as possible of the creaming quality of the fat, but is compelled to depend more and more upon baking powder or other mixtures which produce gaseous compounds upon heating, to leaven the mass, as the amount of shortening is reduced.

Another effect of the fat in cake, along with that of tenderness is in the enhanced keeping quality or apparent freshness. A cake with plenty of good fat in it does not appear or taste as dry as one with less fat.

When we pass over to the cookie type of cake we add a few more problems to the use and action of the fats. In this type we may include the problems of the biscuit and cracker manufacturer.

In the great variety of small cakes known as cookies or biscuits, probably the one most important

function is to produce tenderness or shortness. There has been some work done on the problem of measuring shortening value, but there is so much concerned in the technique of such an investigation that there still remains much to be done. When an investigation is completed on one type of biscuit the question arises as to whether these results fit other types and conditions of production.

The cookies of the small shop have many of them, complicated formulæ in which the shortening plays a minor part. There are the sugar and spice cookies of an infinite variety and the shortening may help in the spread of some, the shape of some and the tenderness of all.

Biscuit and Crackers

In the biscuit plant there are a few classes which may be distinguished.

Take the cakes of the wafer type. These are made with a soft batter, which can be dropped through the die of a machine and cut with a wire, falling upon the pan moving below on a continuous belt, and are put right into the oven. The shortening has a distinct effect upon the character of this piece. If the baker takes advantage of the creaming quality of the shortening, he gets a very different dough and cookie than when he throws in a soft oily shortening and the other ingredients. We have seen the results of such difference not only in the finished cake but in added difficulty and labor cost of handling.

Take another type of biscuit known as short bread. This is a much stiffer dough but in mixing it is possible to make use of the consistency of the shortening. In this type the consistency as well as the chemical composition probably has much to do with the tenderness or shortness of the cookie. This stiff dough is frequently run by forcing it into molds on a roller in such a way as to take the imprint of the mold, and then a flat steel band pulls the shaped dough out and deposits it on the pan.

There are the hard sweets in which the stiff dough is rolled and compressed to produce a close compact mass before being run out in a thin sheet and stamped or embossed with the name and design. Some of these doughs are terribly abused before being baked by being run several times through heavy rollers or breaks. In these the chief function of the shortening is to keep them tender, but its ability to stand the working and rolling without squeezing out is of importance. The character of the shortening also probably plays an important part in the finish of this type of goods.

The biscuit may be followed in this way through a gradual reduction in sugar content, and we finally reach the unsweetened cracker, frequently called the soda cracker. Without touching on the intermediate lines the soda cracker presents an interesting problem in the use of shortening. This, like bread, is a yeast-raised dough, but it takes a much longer fermentation and a higher percentage of shortening.

There is from 8 to 12 per cent of shortening in a soda cracker ordinarily, depending upon the quality of the cracker. Upon this shortening depends the tenderness of the cracker to a marked degree, although this may be influenced by the time and temperature of fermentation and the character of the flour. The doughs are generally rather stiff and the care with which the shortening is incorporated has an influence upon its proper functioning.

Olive Oil Analytical Method*

The Use of the Ultra-Violet Ray in the Detection of Refined in "Virgin" Olive Oil

BY SIDNEY MUSHER

IT becomes a matter of considerable concern to manufacturers of food products of high quality when they are forced into competition with cheaper adulterated or misbranded articles for which no distinct test is acknowledged.

The unparalleled use of renovated olive oil labeled as "virgin" olive oil results, perhaps, from the recognition of some merchants that there is in this country practically no effort devoted to the investigation of physical or chemical tests for the examination of olive oils along these lines.

Attached to this report is a list of articles concerning the use of the ultra-violet lamp for detecting the presence of refined olive oils in "virgin" olive oil.

From the beginning of the experimental work in 1925 to the present time, we consider that there has been practically no disagreement as to the advisability of accepting this test as definite and accurate, just as today the oil merchants of Nice and Marseilles are permitted the use of the lamp by the government in accepting or rejecting shipments of oil which do not approach the standard.

The practicability of the test is further deemed acceptable because in no less than ten experimental laboratories where this work has been carried on, its conclusions have distinctly shown the test accurate in every case.

We are responsible for bringing this test to the attention of Dr. K. S. Gibson of the Bureau of Standards—a report of whose experimental work was given at one of the previous meetings of this Society; but to all practical purposes there was no further research done, in spite of the apparent importance of the use of the ultra-violet lamp, both in the examination of refined and "virgin" olive oils and in the detection of admixtures of other refined vegetable oils with "virgin" olive oil.

Our own experimental work has been confined to clearing the few doubtful points which have arisen in the minds of the researchers over the past few years. For instance, Marcille in the Annales des Falsifications, Volume 21, page 189, reports that he believes the change of fluorescence of olive oils is due to the oxidation fixation properties of the unsaturated fats.

In the course of our experimental work we compared "virgin" olive oil which had remained in a tin from 1913 to the present time, whose free fatty acid was 3.74 per cent and which was distinctly rancid, with other oils. It was found that the fluorescence of this oil was absolutely the same as that of a fresh "virgin" oil sample of this year's crushing. It was also determined that the "virgin" olive oils of different origin showed no change in fluorescence; that is, the Tunisian, French, Spanish, and Italian oils acted in a practically identical manner. We likewise found that the procedure was greatly facilitated by the white background before the lamp.

* Presented at Second Fall Meeting, American Oil Chemists' Society.

We might further mention that the sample of olive oil taken to the Bureau of Standards on October 3, 1927 (crushed during the preceding December), when viewed at the Bureau around October 15, 1928 (or close to two years from the date of its original pressing) showed no change from the original fluorescence, despite the fact that it had suffered distinct deterioration in quality.

Mixtures of refined and virgin oils placed at the disposal of the Bureau by ourselves likewise showed no change in fluorescence, and enabled us to distinguish between the samples of pure "virgin" and mixtures of "virgin" with refined olive oils.

We merely want to offer our suggestion that we believe Marcille has been misled in assuming that oxidation plays a distinct role in the blue fluorescence of a refined oil. After all, we are concerned only with the edible olive oils—"virgin" or renovated—and this experimental work did not take us into the influence of oxidation of oils beyond the time it required to render them rancid or unpalatable.

We therefore are suggesting to the American Oil Chemists' Society that they undertake the work of substantiating the reports of the ten chemists under whose direction this test has been given a place of importance in the Mediterranean countries—where far more considerable quantities of olive oil are used and merchandised, than anywhere else—and especially inasmuch as olive oil is one of the vital industries of those countries.

We are at the present time engaging in further research to more thoroughly substantiate the results already obtained although this lapse of time should not indicate any doubt as to the practicability of the test.

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The American Oil Chemists' Society

Notes and Correspondence

The American Oil Chemists' Society Report of Olive Oil Committee for the Fall Meeting in New York City

The members of the Olive Oil Committee are as follows:

Charles V. Bacon, H. S. Corman, Wallace H. Dickhart, George S. Jamieson, M. F. Lauro, Sidney Musher, C. J. Post, Madison L. Sheely, Ralph W. Wechsler, and Louis M. Roeg, Chairman.

The Chairman has mailed to these members the following plans:

—We have before us the problem of differentiating between Virgin Olive Oil and Refined Olive Oil by the identification of minute quantities of soap in Refined Olive Oil and the absence of soap in Virgin Olive Oil.

In our laboratory (Brewer & Company, Inc., Worcester, Mass.) we have taken a quantity of Virgin fruity Spanish Olive Oil from an original drum. This drum was a part of a shipment imported direct by us last February. The free fatty acid when tested this month was 0.49%.

We refined one gallon of this Virgin Olive Oil using 32° Baumé Solution Sodium Hydroxide, mixing at room temperature for fifteen minutes; raising the temperature to 120°F. in fifteen minutes; and continuing the stirring for an additional fifteen minutes. We had the desirable "break" of the soap from the oil. The next morning we filtered the Olive Oil through fine white filter paper, discarding the first portion to be assured that the Olive Oil was free from suspended soap. The free fatty acid was found to be nil.

This Refined Olive Oil was then tested for the presence of Sodium by extracting the dissolved soap from a 100 cc sample with 25 cc of neutral alcohol. This alcohol was prepared by making slightly alkaline to phenolphthalein with N/50 Sodium Hydroxide and distilling.

This alcoholic extract was filtered, and examined by means of the spectroscope. A loopful on a platinum wire was evaporated by holding near the burner, and was then placed in the flame. The characteristic yellow Sodium band was visible for 1.6 seconds using a stop watch.

When this test was conducted in the same manner with a similar alcoholic extract from the original Virgin Olive Oil, we failed to observe a definite yellow Sodium band, although there was noticed a flash which took only 0.3 seconds to record with the stopwatch. On repeating this test several times we obtained identical results, proving to our satisfaction that the flash cannot be confused with the definite Sodium test.

We also tested these alcoholic extracts by a Micro-Chemical procedure. The extracts were made slightly acid to litmus paper using Hydrochloric Acid, evaporated to dryness on a steam bath, and the residue taken up with 10 cc distilled water and filtered through paper previously washed with distilled water.

One drop of this distilled water solution prepared from the Refined Olive Oil alcoholic extract was evaporated on a slide, and exam-

ined under the microscope, where crystals having the characteristic structure of Sodium Chloride were seen.

One drop of this distilled water solution prepared from the Virgin Olive Oil alcoholic extract when evaporated under similar conditions showed no crystals of Sodium Chloride. However, there were observed green crystals which we believe to be Ethyl-Chlorophyll crystals (Chemistry of Plant Life, Thatcher, 1921, Page 104). These green crystals were absent from the distilled water solution prepared from the Refined Olive Oil alcoholic extract, indicating that the Chlorophyll was removed in refining with Sodium Hydroxide.

Samples have been mailed to the nine members of this committee for corroborating these tests, and their results will be given at our next meeting.

LOUIS M. ROEG,
Chairman.

Italian Olive Oil Market

The olive oil market, according to a commercial newspaper of Messina, Sicily, dated October 8, after a long period of calm is now manifesting some reanimation, especially in southern Italy and Sicily. Prices generally are firm with upward tendency. In Tuscany prices are on the basis of 800-900 lire per quintal and even touch 1050 lire for the best qualities from famous districts. In Umbria and Lazio prices range from 780 to 900 lire according to quality; in Puglia from 750 to about 850 lire; in Basilicata and Algeria the market continues rather calm and transactions are effected on the basis of 815 to 860 francs per quintal, according to a report to the United States Department of Commerce, dated October 12, 1928.

Forecast of Olive Oil Production in Spain

The International Institute of Agriculture in Rome states that the forecast of olive oil production in Spain this year will be about 27.8 per cent of that of last year, or in figures, the 1928 production will be 1,850,000 quintals as compared to 6,656,000 quintals of last year. This is the smallest yield in Spain in the last twenty-years, except in 1910-1911 and 1912-1913 when the yield was 1,085,000 and 630,000 respectively. A large carry-over of the 1927-1928 crop however will prevent to some extent the high prices that might be expected.

The above information was contained in a letter received in the United States Department of Commerce, October 12, 1928, from American Commercial Attache Mitchell, Rome, Italy.

New Margarine Factory at Moscow, Russia

It is reported that the Russian oils and fats industry is working out a project for a margarine factory in Moscow, the capacity of which will be twenty tons per seven hour shift. It is to be constructed on the ground next to the cold storage plant now being built by the Centrosoyous, according to a report received in the United States Department of Commerce from Assistant Trade Commissioner Frank Messenger, London, England.

John P. Harris, Chicago, a representative of the Industrial Chemical Sales Co., Inc., 200 Fifth Avenue, New York City, has moved his headquarters to the Engineering Building, Suite 1306, 205 W. Wacker Drive, Chicago.

Third Quarter Census Report

Washington, D. C., Nov. 5, 1928. —The Department of Commerce announces that the factory production of fats and oils (exclusive of refined oil and derivatives) during the three-month period ended September 30, 1928, was as follows: Vegetable oils, 426,345,582 pounds; fish oils, 43,784,536 pounds; animal fats, 426,972,820 pounds and greases, 84,399,463 pounds; a total of 981,502,401 pounds. Of the several kinds of fats and oils covered by this inquiry, the greatest production, 324,494,084 pounds appears for lard. Next in order is cottonseed oil with 158,704,329 pounds; linseed oil with 141,888,625 pounds; tallow with 100,871,840 pounds; coconut oil with 82,269,151 pounds and corn oil with 25,820,558 pounds.

The production of refined oils during the period was as follows: Cottonseed, 107,496,257 pounds; coconut, 73,388,696 pounds; peanut, 2,146,818 pounds; corn, 20,303,821 pounds; soya-bean, 1,468,157 pounds; and palm-kernel, 1,669,458 pounds. The quantity of crude oil used in the production of each of these refined oils is included in the figures of crude consumed.

The data for the factory production, factory consumption, imports, exports, and factory and warehouse stocks of fats and oils and for the raw materials used in the production of vegetable oils for the three-month period appear in the following statements:

PRODUCTION, CONSUMPTION AND STOCKS OF FATS AND OILS

(In some cases, where products were made by a continuous process, the intermediate products were not reported.)

Kind	Factory operations for the quarter ended Sept. 30, 1928		Factory and Warehouse stocks Sept. 30, 1928
	Production (pounds)	Consumption (pounds)	(pounds)
VEGETABLE OILS:[1]			
Cottonseed, crude	158,704,329	117,781,923	67,951,434
Cottonseed, refined	107,496,257	308,775,901	159,629,289
Peanut, virgin and crude	1,625,724	2,595,495	850,376
Peanut, refined	2,146,818	2,574,006	1,630,554
Coconut, or copra, crude	82,269,151	143,556,886	63,213,908
Coconut, or copra, refined	73,388,696	66,833,914	12,942,497
Corn, crude	25,820,556	26,022,624	16,651,524
Corn, refined	20,303,821	3,222,207	9,588,168
Soya-bean, crude	788,503	3,418,544	5,567,413
Soya-bean, refined	1,468,157	1,040,229	1,736,324
Olive, edible	.....	515,090	3,879,540
Olive, inedible	.....	1,327,301	1,624,499
Sulphur oil, or olive foots	.....	8,603,498	8,518,766
Palm-kernel, crude	.....	10,080,164	6,566,327
Palm-kernel, refined	1,669,458	2,741,267	439,369
Rapeseed	.....	4,132,329	4,627,923
Linseed	141,888,625	136,373,151	121,276,322
Chinese wood or tung	.....	24,696,406	21,177,616
Chinese vegetable tallow	.....	1,046,680	1,661,371
Castor	14,432,375	6,751,898	7,141,931
Palm	.....	48,280,980	27,329,085
All other	816,319	2,541,417	2,904,870

Kind	Factory operations for the quarter ended Sept. 30, 1928		Factory and Warehouse stocks Sept. 30, 1928 (pounds)
	Production (pounds)	Consumption (pounds)	
FISH OILS:[1]			
Cod and cod-liver	381,735	3,799,960	6,485,491
Menhaden	15,810,428	14,934,623	12,879,464
Whale	6,629,250	12,591,247	55,870,199
Herring, including sardine	19,637,580	12,445,059	22,513,346
Sperm	489,375	172,806	3,866,621
All other, including marine animal	836,168	813,265	8,397,608
ANIMAL FATS:			
Lard, neutral	8,443,888	6,394,928	5,467,712
Lard, other edible	316,050,196	4,464,663	98,758,886
Tallow, edible	10,035,932	7,919,871	2,572,727
Tallow, inedible	90,835,908	127,183,975	56,004,608
Neat's-foot oil	1,606,896	1,343,770	1,324,279
GREASES:			
White	13,098,808	10,255,697	5,681,610
Yellow	15,716,123	12,688,409	7,346,984
Brown	11,063,564	15,080,522	5,514,764
Bone	6,086,574	38,965	1,896,750
Tankage	12,338,370	327,586	2,266,209
Garbage or house	21,296,428	24,076,783	7,589,631
Wool	1,424,642	1,605,815	3,478,866
Recovered	787,028	813,763	614,557
All other	2,587,926	1,148,404	2,450,536
OTHER PRODUCTS:			
Lard compounds and other lard substitutes	302,208,496	129,809	23,538,687
Hydrogenated oils	136,918,846	131,496,217	11,818,872
Stearin, vegetable	2,867,079	5,272,427	2,410,063
Stearin, animal, edible	14,033,785	14,208,080	3,604,409
Stearin, animal, inedible	4,718,892	7,573,208	4,734,032
Oleo oil	30,385,393	11,743,425	14,008,735
Lard oil	7,578,430	3,772,006	4,499,543
Tallow oil	3,064,059	2,336,615	1,640,651
Fatty acids	33,066,525	33,794,066	4,725,127
Fatty acids, distilled	13,194,628	11,383,635	2,994,334
Red oil	15,317,679	5,546,000	9,966,868
Stearic acid	10,529,758	3,076,091	3,778,582
Glycerine, crude 80% basis	31,219,604	33,706,434	20,853,692
Glycerine, dynamite	10,321,720	6,642,032	13,720,292
Glycerine, chemically pure	15,739,372	1,532,732	7,280,470
Cottonseed foots, 50% basis	20,666,354	22,656,497	29,768,793
Cottonseed foots, distilled	28,195,392	25,175,801	7,159,566
Other vegetable oil foots	18,960,791	13,646,837	2,110,631
Other vegetable oil foots, distilled	332,677	487,639	462,769
Acidulated soap stock	16,527,864	13,424,099	5,041,567
Miscellaneous soap stock	263,273	424,383	400,491

[1] The data of oils produced, consumed, and on hand by fish oil producers and fish canners were collected by the Bureau of Fisheries.

RAW MATERIALS USED IN MANUFACTURE OF VEGETABLE OILS

Kind	Tons of 2,000 pounds: Consumed July 1 to Sept. 30	Tons of 2,000 pounds: On hand Sept. 30	Kind	Tons of 2,000 pounds: Consumed July 1 to Sept. 30	Tons of 2,000 pounds: On hand Sept. 30
Cottonseed	527,908	566,530	Flaxseed	212,882	103,206
Peanuts, hulled	1,611	529	Castor beans	16,219	6,508
Peanuts, in the hull	914	208	Mustard seed	403	1,289
Copra	64,582	14,413	Soya-beans	2,827	5
Coconuts and skins	669	176	Other kinds	1,136	1,329
Corn germs	43,415	267			

IMPORTS OF OIL SEEDS, QUARTER ENDED SEPT. 30, 1928

Kind	Tons	Kind	Tons
Castor beans	19,404	Popy seed	382
Copra	64,879	Perilla and sesame seed	1,448
Flaxseed	106,570	Other oil seeds	2,464

IMPORTS OF FOREIGN FATS AND OILS, QUARTER ENDED SEPT. 30, 1928

Kind	Pounds	Kind	Pounds
Tallow, edible	834,159	Palm oil	48,145,925
Other animal oils and fats, edible	324,325	Palm-kernel oil	14,557,435
Whale oil	340,222	Sesame oil	1,938,481
Cod oil	2,586,412	Vegetable tallow	43,041
Cod-liver oil	3,835,342	Vegetable wax	834,911
Other fish oils	7,302,502	Cornauba wax	1,477,064
Wool grease	2,347,139	Peanut oil	591,646
Grease and oils, n. e. s. (value)	$49,894	Raps (colza) oil	3,914,228
Olive oil, edible	18,994,381	Linseed oil	35,138
Chinese wood oil or nut oil	34,422,917	Soya-bean oil	4,092,836
Coconut oil	66,217,999	Perilla oil	1,007,844
Sulphur oil or olive foots	11,005,157	Other expressed oils	2,083,995
Other olive oil, inedible	1,154,968	Glycerine, crude	675,576
		Glycerine, refined	191,634

EXPORTS OF FOREIGN FATS AND OILS, QUARTER ENDED SEPT. 30, 1928

Kind	Pounds	Kind	Pounds
Animals oils and fats, edible	135	Cocoanut oil	513,398
Fish oils	3,975	Palm and palm-kernel oil	274,981
Other animal oils and fats, inedible	182,223	Peanut oil	7,888
Olive oil, edible	15,041	Soya-bean oil	496,520
Chinese wood oil or nut oil	1,953,835	Other expressed oils	39,489
		Vegetable wax	188,368

EXPORTS OF DOMESTIC FATS AND OILS, QUARTER ENDED SEPT. 30, 1928

Kind	Pounds	Kind	Pounds
Oleo oil	16,180,590	Other animal greases and fats	14,239,889
Oleo stock	1,684,870	Cottonseed oil, crude	1,793,680
Tallow	1,283,854	Cottonseed oil, refined	2,412,648
Lard	149,755,985	Corn oil	91,572
Lard, neutral	4,894,719	Vegetable oil lard compounds	1,339,863
Lard compounds, containing animal fats	973,008	Other edible vegetable oils and fats	958,065
Oleo and lard stearin	1,307,278	Coconut oil	7,071,459
Neat's-foot oil	270,910	Linseed oil	512,339
Other animal oils, inedible	177,924	Soya-bean oil	1,627,152
Fish oils	110,228	Vegetable soap stock	1,768,761
Grease stearin	478,953	Glycerine	497,119
Oleic acid, or red oil	1,387,747		
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Of OIL & FAT INDUSTRIES, published monthly, at New York, N. Y., for October 1, 1928.

State of New York)
County of New York) ss.:

Before me, a Notary Public in and for the State and county aforesaid, personally appeared Alan Porter Lee, who, having been duly sworn according to law, deposes and says that he is the Editor of the OIL AND FAT INDUSTRIES, and that the following is, to the best of his knowledge and belief, a true statement of the ownership, management (and if a daily paper, the circulation), etc., of the aforesaid publication for the date shown in the above caption, required by the Act of August 24, 1912, embodied in section 411, Postal Laws and Regulations, printed on the reverse of this form, to wit:

1. That the names and addresses of the publisher, editor, managing editor, and business managers are: Publisher, Leod D. Becker, 420 Madison Ave., New York City; Editor, Alan Porter Lee, 420 Madison Ave., New York City; Managing Editor, none; Business Managers, Leod D. Becker, 420 Madison Ave., New York City.

2. That the owner is: (If owned by a corporation, its name and address must be stated and also immediately thereunder the names and addresses of stockholders owning or holding one per cent or more of total amount of stock. If not owned by a corporation, the names and addresses of the individual owners must be given. If owned by a firm, company, or other unincorporated concern, its name and address, as well as those of each individual member, must be given.) Russell Palmer, 71 Columbia St., Seattle, Wash.

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5. That the average number of copies of each issue of this publication sold or distributed, through the mails or otherwise, to paid subscribers during the six months preceding the date shown above is: (This information is required from daily publications only.)

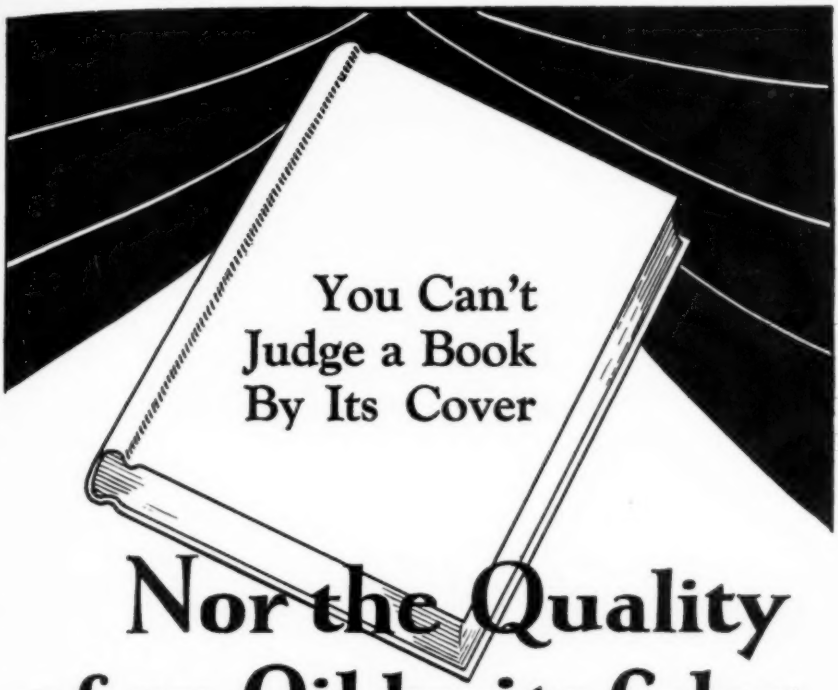
ALAN PORTER LEE, Editor.

Sworn to and subscribed before me this 29th day of September, 1928.

(Seal) Johanna E. Schult, Notary Public.

N. Y. Reg. No. 0-919. N. Y. County Clerk's No. 1326. Queens County Clerk's No. 2339. Reg. No. 2601.

(My commission expires March 30th, 1930.)



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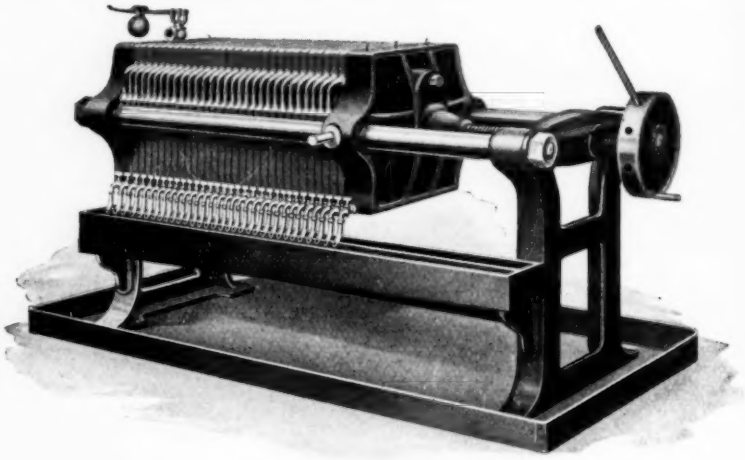

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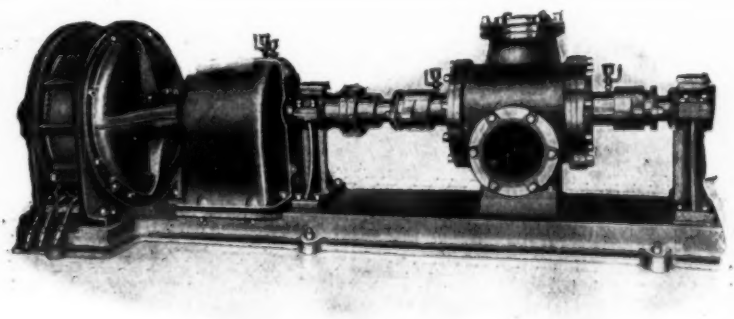

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